# MICROFOCUS RADIOGRAPHY

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Thank you very much.

I should emphasize that what I am going to talk about is really the result of a very brief program which was funded at Harwell by David Godfrey of AMTE, who is probably well-known to ceramic practitioners here. We were very glad to have this contact because it gave us access to the TTCP specimens which were prepared in this country; specimens, in particular, of silicon nitride tiles containing seeded defects, which many of you have studied by various techniques.

My collaborator was Ron Smith in this work. The objective was to compare the sort of ultrasonics that we had with the performance of the radiographic facilities for looking at these very small seeded defects.

Now, I was thinking that my talk would come at the end of this session and, therefore, that you would already be familiar with the types of defects which have been introduced in these tiles and their sizes, but I am afraid I do not really have time in my talk to discuss all that. I imagine that subsequent speakers will be giving you details of that.

The ultrasonics that we used was basically a conventional C-scan using a plane 25-megahertz probe and the usual sort of loss of back-echo technique to see what defects we could find. And naturally, with such a comparatively unsophisticated system, we could see some of the defects, but by no means all of them.

We then went on to the radiography. The particular sort of radiography that we are doing involves the use of a microfocus source. This is now commercial equipment at home, and I am afraid I cannot tell you all the present details of it, but the vital thing about it is that this instrument called the E12 has a 15-micron diameter source of very bright x-rays. This particular manifestation of the instrument operates from 30 to 80 kilovolts. There is another instrument which operates at lower voltage.

Now, when one has this microfocus source, there is great advantage in using a projection system whereby the specimen is quite near the source and the recording plate is a good deal further away, so that one obtains, effectively, a magnification. For reasons which are not obvious, but which can now be justified, there is an improvement in the contrast and resolution in the use of a system of this kind by comparison with what you can get with a contact radiograph.

My colleague, Ron Smith, has recently carried out a Monte Carlo computer calculation showing how scattered radiation in this projection case can be lost entirely to the recording plate; and therefore, the contrast and the resolution of small defects, is improved by comparison, as I say, with the contact radiograph. That paper has been submitted to the British Journal of Nondestructive Testing and should be available at anytime in the next month or two.

What I should just briefly like to do now is show you exactly the numbers that we obtained.

In the first viewgraph (Table 1), for which we must thank our hosts here for producing with their customary generosity and the efficiency we have come to take for granted, at a moment's notice,let me say.

We can see this refers to the material as hot pressed silicon nitride. We have looked up the best available figures that we could find for the density and the acoustic impedance properties, and you can see along the top line there what these numbers work out for each material. From this we then calculated the reflection coefficient and what we thought was possibly of more direct importance, the transmission coefficient, first of all, from the silicon nitride into the defect material and from the defect material back to the silicon nitride, and the final column shows the ultimate transmission through a system of that kind.

We thought, perhaps, either the reflection coefficient or the final products, D1D2 might be the more effective indicator of how likely it was that you could find a given defect by ultrasonics. Of course, the first column, the density, gives a good idea of how likely it is that you will find the same defects by radiography.

The difficulty lies, of course, in the range of silicon carbide, silicon and graphite. We refer to our Chairman up here, I think; his well-known work on the fracture toughness of ceramics strongly suggests that it is materials of this kind which themselves have low fracture toughness and low modulus which may be the most dangerous.

The figures have also been worked out for reaction-bonded silicon nitride (Table 2). And there, again, they may be slightly different, but not substantially. We put in the two cases for carbon. We do not know quite what form the carbon or graphite inclusions might be when they are actually in the ceramic tile. But you can see that when D1D2 approaches unity, it looks pretty unlikely that you would be able to find it by ultrasonics. But when the figure is substantially different from unity, then, of course, there is a very good chance. Of course, the density figures also show some of the impurities will come out as light patches and some of them will come out as dark patches.

Table 1

HPSN: Density and Acoustic Properties of the Inclusion Materials

| Material | Density kg $m^{-3}$ | Longitudinal Velocity km/s | Acoustic Impedance $kg/m^2s$ | Reflection Coefficient R | Transmission Coefficient $Si_3N_4$-to-defect $D_1$ | Transmission Coefficient Defect-to-$Si_3N_4$ $D_2$ | $D_1D_2$ |
|---|---|---|---|---|---|---|---|
| HPSN | 3.18 $10^3$ | 10.5 | 3.3 $10^7$ | 0.00 | 1.00 | 1.00 | 1.00 |
| WC | 15.77 $10^3$ | 6.6 | 10.4 $10^7$ | 0.52 | 1.52 | 0.48 | 0.73 |
| Fe | 7.87 $10^3$ | 5.9 | 4.6 $10^7$ | 0.16 | 1.16 | 0.84 | 0.97 |
| BN | 2.25 $10^3$ | 5.5 | 1.2 $10^7$ | -0.47 | 0.53 | 1.47 | 0.78 |
| SiC | 3.22 $10^3$ | 12.0 | 3.8 $10^7$ | 0.08 | 1.08 | 0.92 | 0.99 |
| Si | 2.33 $10^3$ | 11.9 | 2.8 $10^7$ | -0.08 | 0.92 | 1.08 | 0.99 |
| C (Graphite) [0001] | 2.26 $10^3$ | 22.7 | 5.1 $10^7$ | 0.21 | 1.21 | 0.79 | 0.96 |
| C (Graphite) ⊥ [0001] | 2.26 $10^3$ | 4.56 | 1.0 $10^7$ | -0.53 | 0.47 | 1.53 | 0.72 |

Table 2

RBSN - Density and Acoustic Properties of the Inclusion Materials

| Material | Density kg $m^{-3}$ | Longitudinal Velocity $km/s^{-1}$ | Acoustic Impedance $kg/m^{-2}\ s^{-1}$ | Reflection Coefficient R | Transmission Coefficient $Si_3N_4$-to-defect $D_1$ | Transmission Coefficient Defect-to-$Si_3N_4$ $D_2$ | $D_1D_2$ |
|---|---|---|---|---|---|---|---|
| RBSN | 2.20 $10^3$ | 6.14 | 1.35 $10^7$ | 0.00 | 1.00 | 1.00 | 1.00 |
| WC | | | | 0.77 | 1.77 | 0.23 | 0.41 |
| Fe | | | | 0.55 | 1.54 | 0.45 | 0.69 |
| BN | | | | -0.06 | 0.94 | 1.06 | 1.00 |
| SiC | | | | 0.49 | 1.49 | 0.51 | 0.76 |
| Si | | | | 0.35 | 1.35 | 0.65 | 0.88 |
| C (Graphite) [0001] | | | | 0.58 | 0.87 | 0.42 | 0.37 |
| C (Graphite) ⊥ [0001] | | | | -0.15 | 0.85 | 1.15 | 0.98 |

Now, this is our final table of results (Table 3). The defects that we claim to see are as follows:

The column labeled "U" refers to defects which we found by our simple C-scan of the sites and which can be seen on the MUFAX four-level recorder. The X radiograph, the defects found by X-rays, were obtained in many cases by projection radiography. Some of the defects could be found with a magnification factor of about four, but the smallest ones, particularly in the case of boron nitride and silicon we could only see by using a magnification factor of 12. Now it is obviously quite impracticable to completely survey an item with a magnification of 12.

Table 3

Summary of Present Results on Silicon Nitride Tiles

| Matrix Material; Defect Type | | 25 μm | Fine 125 μm | Medium 250 μm | Coarse 510 μm |
|---|---|---|---|---|---|
| NC-132 HPSN; | Fe | X, U | X, U | X, U | X, U |
| | WC | X | X | X, U | - |
| | Si | | X | X | X |
| | SiC | | | | X |
| | BN | X, U | X, U | X, U | X, U |
| | C | | | X | X, U |
| NC-320 RBSN; | Fe | - | X, U | X, U | X, U |
| | Si | - | | X | X, U |
| | SiC | - | | X | X, U |
| Low Density | | - | | | X |
| | C | - | U | X | X |
| Pores | | - | X | X, U | X |

Relative effectiveness of detection of seed defect inclusions by ultrasonicsand radiography.

X = radiography
U = ultrasonics

It is a very big microscope job, and to do X-ray microscopy with a large magnification factor is not very practicable. But what we did find is that using our own ultrasonic system and carrying out a B-scan, recording a B-scan along a line in the tile on which we had reason to suppose there were defects, we could identify areas which aroused suspicion, and we would then carry out the projection radiography on those particular areas.

In particular, we were able to find, as we say here, very small defects in the hot pressed silicon nitride. In the reaction bonded silicon nitride, we could not do quite so well, obviously, because there is more background scatter or more noise in the system.

On the whole, we think that these figures are substantially better than others which have been claimed by people elsewhere who have used other systems of radiography. We are therefore suggesting that radiography has an important part to play, in conjunction, you understand, we would suggest, in this important problem of detecting and classifying defects in silicon nitride.

Thank you.

## SUMMARY DISCUSSION

Bruce Thompson (Rockwell Science Center [now Ames Laboratory]): Perhaps we should take a moment or two to entertain any questions on Bill's work before I proceed.

Bill Reynolds (AERE Harwell): I should say I have a copy of the report here. And the people who are interested in the details, I can show you some prints of the radiograph, but I haven't got any slides prepared.

James Aller (NSF): How long was your exposure on the film?

Bill Reynolds: Fairly long. I think of the order of - anything up to two hours.

Unidentified Speaker: Can you describe the microfocus unit a little bit better?

Bill Reynolds: I am afraid I cannot. It has had a long history. Originally the microfocus system depended on a specially developed electrostatic focus, but since then, the whole thing has been taken over by a commercial organization. I do not know exactly what they have done with it. We just use it as a very valuable source of very fine focus x-rays.

Unidentified Speaker: Is it done in vacuum or -

Bill Reynonds: Yes, it's continuously pumped.

P.S. Ong (University of Houston): What spot size, curve, and voltage? Do you have any information about spot size, curving, and voltage of the x-ray tube?

Bill Reynolds: The voltage we used in this particular case was 80 kilovolts; the spot size is 15 micron diameter; and this is continously pumped.

P.S. Ong: Have you ever considered using an image intensifier at all?

Bill Reynonds: We have, in fact, done that. I may say that this is the least important application of the system that we have.

Bruce Thompson: Thank you very much for giving that very interesting but impromptu presentation.